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Design of a Laboratory for Particulate Analysis

United States Steel Corp.
Monroeville, PA

Prepared for

Environmental Monitoring Systems Lab.
Research Triangle Park, NC

May 81

U.S. Department of Commerce
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R. J. Lee, J. S. Lally, and R. M. Fisher.

PERFORMER: United States Steel Corp., Monroeville, PA.
Research Lab.
Contract EPA-68-02-3247

SPONSOR: Environmental Monitoring Systems Lab., Research Triangle Park, NC.
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KEYWORDS: *Air pollution, *Particles, *Laboratory design, *Asbestos.

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DESIGN OF A LABORATORY FOR PARTICULATE ANALYSIS

by

R. J. Lee, J. S. Lally and R. M. Fisher
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FORWARD

Measurement and monitoring research efforts are designed to anticipate potential environmental problems, to support regulatory actions by developing an in-depth understanding of the nature and processes that impact health and the ecology, to provide innovative means of monitoring compliance with regulations, and to evaluate the effectiveness of health and environmental protection efforts through the monitoring of long-term trends. The Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, has responsibility for: assessment of environmental monitoring technology and systems; implementation of agency-wide quality assurance programs for air pollution measurement systems; and supplying technical support to other groups in the Agency including the Office of Air, Noise, and Radiation, the Office of Toxic Substances, and the Office of Enforcement.

The measurement of asbestos in environmental matrices including airborne particulate matter has as yet proven a challenge to the scientific community, including workers in EPA.

As part of the ongoing efforts of the Environmental Monitoring Systems Laboratory, this study was authorized by me to obtain estimates of resources necessary to staff and equip a laboratory to provide EPA with a laboratory to serve as a focal point to advance the EPA in its objective of understanding the effects in the biosphere resulting from environmental contaminants from components of particulate matter.

T R Hauser

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PREFACE

The assessment of air quality and other environmental factors for effects on health and welfare, control strategy, standards setting and compliance, all demand accurate validated data obtained from environmental monitoring. All of these endeavors, especially relating health effects to environmental levels, have suffered from the lack of proven monitoring methodologies for the identification, characterization, and physical and chemical quantification of the different species of asbestos.

To strengthen the nation's capability for environmental research, the EPA commissioned the design of a particulates analysis laboratory, with an emphasis on asbestos, to serve as a model facility in terms of instrumentation, staff, methodology, and credibility. Specific objectives of the assignment were "development of specifications and recommendations for a particulates analysis laboratory with special emphasis on the identification and measurement of dimensions and concentration of serpentine and amphibole asbestos. Specimen preparation methods, conditions necessary to maintain adequate clean room standards, equipment for optical microscopy, x-ray diffraction, electron microscopy, electron-diffraction, and computer data processing are to be evaluated and prescribed, and the number, qualifications, and availability of all personnel necessary to supervise and operate the facility defined."

This report addresses the EPA needs for a particulates analysis laboratory and its requirements and contains detailed recommendations (based on extensive experience in this area by U. S. Steel Research Laboratory) concerning the design, staff, instrumentation, methodology and management of the facility. In addition to utilization of customary light and electron optical methods, the adoption of newly developed techniques for automatic measurement of particle size, shape, and composition is highly recommended. These methods have been in use in the U. S. Steel Research Laboratory for three years and have proven to be rapid, accurate and consistent for the characterization of particulates in a wide variety of samples.

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ABSTRACT

The effects on human health resulting from asbestos exposure in various environments are a subject of national concern and debate. There is a pressing need for better information about the concentration of asbestos in our environment, and the importance of the size/composition/shape of asbestos fibers in causing adverse health effects. Yet, in general, current asbestos-analysis methods are time consuming procedures of questionable reliability. Unfortunately, no laboratory exists that was designed, equipped, and most importantly, staffed to perform asbestos analysis.

In recognition of the need for a "state-of-the-art" laboratory for particulates analysis, with emphasis on asbestos, the Environmental Protection Agency commissioned the design of a suitable facility and the development of recommendations for equipment and personnel. In this report the need for such a facility is reviewed; the considerable investment in equipment and large annual operating expenses are justified, and a conceptual framework for a particulates analysis laboratory is formulated. The basis for selection of optimum equipment, the results of a detailed survey of equipment manufacturers, the design layout of the proposed laboratory, the availability of skilled personnel, and a review of analysis methodology are summarized.

It is concluded that the nature of current and past problems related to particulates analysis is such that these problems can best be resolved by organizing the laboratory as a "center of excellence." Functions of the laboratory should include methods development, training, refereeing, standard sample development and evaluation, and the analysis of difficult samples. It is recommended that an advisory panel of experts be established to provide on-going peer review of the scientific quality and effectiveness of these activities.

To fulfill such a broad commitment to excellence it is imperative that the laboratory be staffed with highly qualified and motivated personnel led by an acknowledged expert in asbestos analysis. Only a very small number of persons have the necessary qualifications for the senior positions. The salary offered as

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well as the caliber of the facility, including the staff and equipment and the degree of autonomy, will be determining factors in attracting one of these individuals to the laboratory.

A staff of 7 to 14 is recommended. The smaller number is the core level necessary to maintain excellence in analytic techniques with a minimal 'service' capability. The optimal staff complement is 14 persons. At this level about 50 percent of the effort should be devoted to "routine" requests for the analysis of special samples requiring a high level of technical expertise. The remaining 50 percent should be reserved for internally developed projects independent of requests for analysis or advisory activities. In addition to recommendations concerning the equipment, staffing, and formation of the proposed facility, specific proposals for expediting start-up, selection of initial projects, and maintaining longer range developments are included in the report.

The initial capital cost for laboratory instrumentation and specialized air-handling equipment is estimated at 0.75 million dollars (excluding the cost of the building) and start-up within 1 year after completion of the building is believed to be achievable. Minimum staffing and an initial level of support of $308,000 per year is recommended to inaugurate the proposed laboratory. At the optimal staffing, an operating budget of $534,000 per year is anticipated, with about $260,000 generated by requests for services from outside groups or agencies.

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LIST OF ABBREVIATIONS AND SYMBOLS

ABBREVIATIONS

AIA	--Automatic Image Analysis
BSE	--Backscattered Electron
EDS	--Energy Dispersive Spectroscopy
EM	--Electron Microscopy
EPA	--Environmental Protection Agency
DBCS	--Digital Beam Control System
GSA	--General Services Administration
HEPA	--High Efficiency Particle Absolute
SAD	--Selected Area Electron Diffraction
LaB_6	--Lanthanum Hexaboride
SEM	--Scanning Electron Microscopy
SE	--Secondary Electrons
STEM	--Scanning Transmission Electron Microscopy
TEM	--Transmission Electron Microscopy
USS	--United States Steel Corporation

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INTRODUCTION

For some time the methodology and results of asbestos analysis, especially by electron microscopy, have been a source of controversy in the scientific community. The highly elongated shape that is an essential physical attribute of all asbestos material has long been recognized as a determining factor in the inhalation of asbestos, and therefore, in the production of adverse health effects in exposed individuals. In the past, morphological identification of asbestos was the only reliable, or necessary, method for monitoring the concentration of asbestos fibers in the workplace. Thus, phase contrast optical microscopy became the primary tool for monitoring asbestos in industrial health studies.

In using this technique the operative assumption is that if a particle is fibrous, it is asbestos. This technique is effective in monitoring concentrations in environments where asbestos is used or known to occur in significant quantity. However, optical microscopy is not reliable for monitoring the concentration of asbestos fibers less than 0.5 μm in diameter and 5 μm in length, or where it occurs in low concentrations. Accurate knowledge about the occurence of particles of these dimensions has become increasingly important as concern has evolved about the potential health effects of suboptical fibers

Tradition and acceptance of optical data on asbestos by the medical community prompted attempts to extend the use of optical microscopy to environmental monitoring. Electron microscopy has the requisite resolution but requires a much higher level of sophistication for sample preparation, counting, and identification of asbestos. Procedures have been developed to permit electron microscope measurement of suboptical particles, but with limited success. As a result, electron microscopy is not generally regarded as a quantitative technique for the measurement of "asbestos" concentration.

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This situation persists, in spite of the expenditure of substantial sums of money by various federal agencies and

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other groups. Recognizing that a significant problem exists, the EPA solicited proposals and subsequently awarded a contract to USS Research to develop a design of a laboratory for particulates analysis, with emphasis on asbestos.

On the basis of our own experience and a survey of research organizations involved in this type of analysis, we have formulated an appropriate laboratory concept, defined the necessary equipment and personnel, and recommended an operational framework, a capital and operating budget, and the analytical methodology.

The report consists of seven semi-independent sections. As indicated in the table of contents, the conceptual framework, rationale, justification, functions, setting, and recommended support levels are described in Section 1. The recommended methodology is discussed in Section 2, with added emphasis on emerging techniques that promise to dramatically reduce the considerable effort and difficulties currently associated with microscopic techniques. In Section 3 the physical plant is described, in Section 4 the selected instruments are discussed, and in Section 5 personnel requirements are specified. Section 6 provides an estimate of initial and annual costs, and the timetable required to bring the laboratory into existence. The recommendations are summarized and some suggestions for initial projects are presented in Section 7. Detailed information useful when the laboratory is to be set up is included in several appendices.

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SECTION 1

PURPOSE AND FUNCTION OF A FEDERAL PARTICULATES ANALYSIS LABORATORY

1.1 Rationale for a Particulates Analysis Laboratory

The size, shape, composition, and abundance of particulates are widely recognized as factors determining the nature of biological effects resulting from their inhalation or ingestion. The ability to reliably characterize and quantify the abundance of relevant species in environmental samples would open the door to improved understanding of their effects on human health, and ultimately to control of their occurrence in the environment through carefully established regulations and monitoring.

Accurate data on levels and type of asbestos are necessary for estimation of the magnitude of any problem (whether it be relating health effects to levels), estimating the health effects problems for risk assessment, the basis for control strategies, for regulatory standards setting, and for compliance. Methodologies suitable for determination of environmental levels for trend assessment, for health effects and other correlations may and probably should not require utilization of methodologies designed to obtain data for purposes of compliance.

Generally, microscopic techniques are required to analyze individual respirable particulates. Unfortunately particulate analysis, including sampling, identification, and concentration measurements while conceptually simple, is an area dominated by uncertainty. For example, the type and dose of asbestos to which humans are exposed are known to be significantly related to response(1). But the specific relationships between size, shape, composition and abundance, i.e. "levels" (1 g) and "kinds" (1 h) of asbestos inhaled or ingested by humans and the production of adverse health effects is uncertain. This problem has been a subject of concern for years.

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In spite of this, no laboratory has developed a reliable and comprehensive method incorporating all aspects of the analysis. Some laboratories have emphasized sample preparation. Others have concentrated on identification. Still others have focussed on counting procedures and statistics. During this period, a number of contracts have been awarded by the EPA for methods development. None of these has resulted in a robust method, capable of reliable, reproducible measurement of asbestos concentrations in environmental samples. In fact most laboratories involved in this work were not equipped or staffed to conduct sophisticated electron microscope and diffraction analysis on a routine basis. These problems hamper the definition and resolution of an important public health question. What are the levels and health effects of asbestos in the workplace, public buildings, homes and other environments?

In this light, the creation of a laboratory devoted to developing and maintaining excellence in the field of particulates analysis, with an emphasis on asbestos, should be of the highest priority. This laboratory should be concerned with monitoring methodological adequacy for all types and phases of particulate monitoring.

1.2 Justification for a Federal Facility

The pressing need for an independent and credible center of excellence for particulates analysis is apparent in light of several recent events. Five examples come to mind.

First, a provisional method for the analysis of airborne asbestos(2) commissioned by the EPA and widely distributed as the provisionally recommended procedure for electron microscopy has been severely criticized by numerous sources even though most of the individual steps in the method represent the state-of-the-art. Specifically, the final procedure recommended a sample-collection procedure generally regarded as unproven, and the identification procedures are unreliable for measuring the amphibole asbestos content of multicomponent samples. For example, a survey of the asbestos content of iron ore bodies in the Mesabi Range, Minnesota was conducted by the contract laboratory that developed the provisional procedure. The data showed amphibole concentrations which differed by less than a factor of two for ore bodies in the extreme Western and Eastern Mesabi. However, in the Western Mesabi ore body, amphiboles do not occur, and in the Eastern Measabi ore body, amphiboles are visible microscopically. This measurement anomaly is not atypical, but has not been resolved.

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Second, the EPA contracted for the development of an interim procedure for the analysis of asbestos in water(3). The development project was thorough and resulted in a procedure which should yield reliable data when carefully used to measure the chrysotile content of water. However, a survey of asbestos in the mining-industry recycle water and effluents(4), for which the interim procedure was used, is being questioned by industry, government, and independent experts.

In this case, the problem is not in the method, but in its use. A review of the data revealed that the contracting laboratory (which was not the laboratory that developed the interim procedure) did not use a quality-control program to determine blank levels and the effects of sample dilution, as recommended by the interim procedure. They reported asbestos concentrations based on fiber counts below those required for statistical purposes by the interim procedure and failed to assess the statistical significance of the data. Because of these factors they reported higher concentrations of asbestos in areas where none occurs geologically than in discharge water from asbestos mines.

Third, a recent EPA water-quality criteria document(5) recommends target control concentrations for the asbestos content that are incompatible with the physical concentrations of asbestos that can be measured reliably. In addition, because of the unresolved problems with the analytical methods, the validity of the data used to calculate the target levels is questionable. Both of these problems could have been avoided if persons with the necessary expertise had been available and requested to review the document.

Fourth, discrepencies were reported in a round-robin analysis of samples collected near a serpentinite rock quarry(6). These differences have essentially been left unresolved because no single laboratory is available that can provide a definitive analysis and comparison of preparation methods.

Fifth, in a recent comparison of the results from 150 samples from New York City area schools, two prestigious laboratories disagreed on whether or not 26 percent of the samples contained asbestos(7).

Thus it is clear that the development of superior techniques for characterizing the quantity, type, and frequency of airborne particulates is of paramount importance. It is also apparent that EPA needs a center of excellence to resolve

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discrepancies of this nature. Until every laboratory presumed to be qualified to identify asbestos, amphiboles, and other mineral particles can produce reliable and credible data, analyses of particulates in the environment will be subject to question and exceedingly expensive. Until reliable data are forthcoming, we will not be able to resolve specific questions about the significance of size, shape, or composition of particulates, especially asbestos, in producing adverse health effects. Furthermore, the establishment of appropriate regulations and monitoring will not be possible.

A specialized laboratory dedicated to the development, evaluation, and promotion of analytic procedures must be the responsibility of the Federal government through one of its administrative agencies. No other organization has the resources to acquire state-of-the-art instrumentation on a continuing basis, assemble a well trained and experienced staff, and commit them to the long-term objective of improving particulates analysis methods to permit the establishment of effective control strategies.

1.3 Function of the Particulates Laboratory

The preceding examples serve to identify several problem areas of current concern, the resolution of which will be one of the primary functions of the particulates analysis laboratory. Perhaps the most obvious role is that of a service facility for routine microscopic analysis, capable of handling a high volume of sample analyses. However, in our view, a single service laboratory could not cope with the anticipated large volume of analyses and fulfill the other commitments without a massive expenditure of funds for adequate staff and equipment. Rather, the primary service function of the laboratory should be that of a center of excellence. As such, it would provide a wide variety of essential services to other laboratories or agencies concerned with the acquisition or interpretation of data about the occurrence of particulates.

Referee - The particulates laboratory would act as a referee for resolving differences between analytical laboratories. It would be capable of performing method checks on techniques developed elsewhere, and would provide a definitive analysis in a referee role when necessary.

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Special Analysis - The laboratory would be capable of analysing unusually difficult samples or samples requiring a very short response time.

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Quality Assurance - The laboratory would develop or evaluate standards, prepare and check split samples for blind audits, make recommendations on new specimen methods and analytical techniques, in collaboration with outside organizations such as the National Bureau of Standards.

In particular, asbestos aerosols and exposures to them have received a great amount of attention in Europe and the United Kingdom. No consideration has been given to this valuable background in describing the laboratory. The work of the U.I.C.C. (Union International Contre le Cancer) in standardizing asbestos specimen and in assessing asbestos aerosols should be recognized and provision made in the laboratory to take advantage of this standardization work and to permit future collaboration.

Contract Monitoring - The laboratory would provide assistance to project offices in establishing the efficiency and accuracy of methods developed as well as results produced by contract laboratories.

Enforcement Support - The laboratory would provide analytical support and technical consultation in enforcement and rulemaking procedures.

Training - The laboratory would act as a center for the training and, particularly, certification of groups or persons performing analyses under EPA contracts. Individuals with prior training in optical or electron microscopy would spend some time at the particulate analysis facility becoming acquainted with recommended methods of specimen preparation, polarizing light microscopy, interpretation of electron diffraction patterns, and other methods of particulate characterization.

Methods Development - Involvement in the preceding functions would provide the laboratory with the experience necessary to set priorities for methods or technique development for support of EPA's monitoring needs. On-going activity in this area would in turn maintain the expertise necessary to perform a viable service to other groups or agencies. The methods projects would include the development and correlation of optical and electron microscopic techniques for characterization of the particulate content of airborne, water, bulk, dust, and biological samples. Major emphasis would be placed on adopting automatic image analysis for scanning transmission electron microscopy and on direct computer interpretation of electron diffraction patterns.

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In each of the examples cited in Section 1.2, the laboratory could have performed a valuable service in one or more of the above capacities, thereby avoiding needless controversy, confusion, or expenditure of funds.

1.4 Setting and Support of the Laboratory

It is presumed that the proposed particulates analysis facility would be located near an existing national laboratory and housed within its administration structure. However, to effectively carry out the multiplicity of functions described and attract the caliber of staff required, the laboratory should be semi-autonomous, staffed with highly qualified specialists setting their own priorites. Every effort must be made to insulate the laboratory from sudden changes in focus or priority, yet to ensure that the laboratory's objectives and projects are responsive to agency needs. We recommend the formation of an advisory panel, composed of knowledgeable persons from government agencies, academic institutions, and industrial laboratories, to provide peer review of programs, evaluate progress, and assist in setting long-term direction, objectives, and goals.

We believe that approximately 50 percent of the operating budget should cover the cost of methods development, participation in collaborative analysis programs, enforcement assistance, technical publications, and travel. The remainder of the support could be derived on the basis of costs of services charged to groups requesting such services.

1.5 Implementation

To meet the needs of the Environmental Protection Agency as defined in the Objectives for the facility considered above and fulfill the various functions described, rather demanding requirements for equipment, personnel, management, and on-going peer review of the activities must be met. These topics are discussed in detail in the following section of this report and specific recommendations are put forward for each of the steps that must be taken to establish and maintain a national center of excellence for studies of particulates that affect the quality of the environment.

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SECTION 2

ANALYSIS METHODOLOGY AND SAMPLE ROUTING

2.0 Introduction

The purpose of the particulates analysis facility is complete and definitive characterization of environmental, primarily air and water, samples to relate to potential health hazards as discussed previously. The needs of the EPA for environmental monitoring and particulates analysis include (1) routine measurements on a large volume of samples with a well-defined origin and type until satisfactory methods are implanted in contract laboratories, (2) one-of-a-kind samples of unknown matrix, (3) evaluation of methods using sample splits prepared for comparative tests by several laboratories or referee analysis for quality control purposes, and (4) critical studies where complete chain-of-custody responsibility must rest with one or two individuals. Each of these are definitive activities requiring different levels of scientific, technical and management expertise to carry them out effectively.

When required, thorough characterization could involve positive identification of all fibers or particles observed, measurement of their concentration, and determination of the distribution of particle dimensions and shapes. It is presumed that the laboratory will be primarily concerned with the more difficult rather than routine samples. A wide variety of sample types must be accommodated including dusts, bulk quantities of building materials, mine or quarry products, suspended particulates in air as collected on filters, particulates in water, and biological specimens. A methods development and evaluation program is imperative if the laboratory is to foster new developments in the field and to maintain and promote an awareness of potential sources of uncertainties or errors.

The following sections on characterization techniques are intended to outline the generalization of the various analytical procedures rather than specific details as might be found in texts or "methodology manuals(8)(9)(10)."

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It must be noted that this report deals only with the processing of samples after they reach the laboratory. Considerable effort should also be placed on reviewing currently available sampling techniques and recommendations on preferred procedures and programs for methods improvement established.

2.1 Analytical Techniques

The several methods of analysis required to fulfill the obligations of the laboratory include light and electron optical techniques ranging from low-magnification binocular screening, phase-contrast and polarized-light microscopy to scanning and transmission electron microscopy with capabilities for x-ray spectroscopy and electron diffraction. For sub-optical microscope particulates the scanning electron microscope (SEM) can provide particle information based on manual characterization of morphology and composition and, with newly developed digital beam-control devices, automatic measurement of particle size, shape, and probable identity at present. The scanning-transmission electron microscope (STEM) will permit conventional manual x-ray and electron-diffraction analysis for asbestos con tent and with the addition of beam-scan-control accessories, as mentioned above, automated particle characterization. The SEM and STEM instruments provide generally similar chemical and size information about particles but on an increasingly finer scale. Correspondingly, an increased level of sophistication is required to operate and maintain the instruments and to interpret the information derived from each.

2.2 Sample Routing

A significant fraction of samples will be generated internally. Also, in the future, particulates other than asbestos can be expected to be of importance. However, in the following, we treat all samples as though they were to be analyzed for asbestos and were originated by outside sources. In general, the words asbestos, fiber, or particulate may be treated interchangeably.

The natural flow of samples after arrival is from preliminary preparation and screening to higher levels of observational detail and information. To this end, the flow of samples through the laboratory is illustrated schematically in Figure 1, and the recommended analyses at each stage are described in the following sections.

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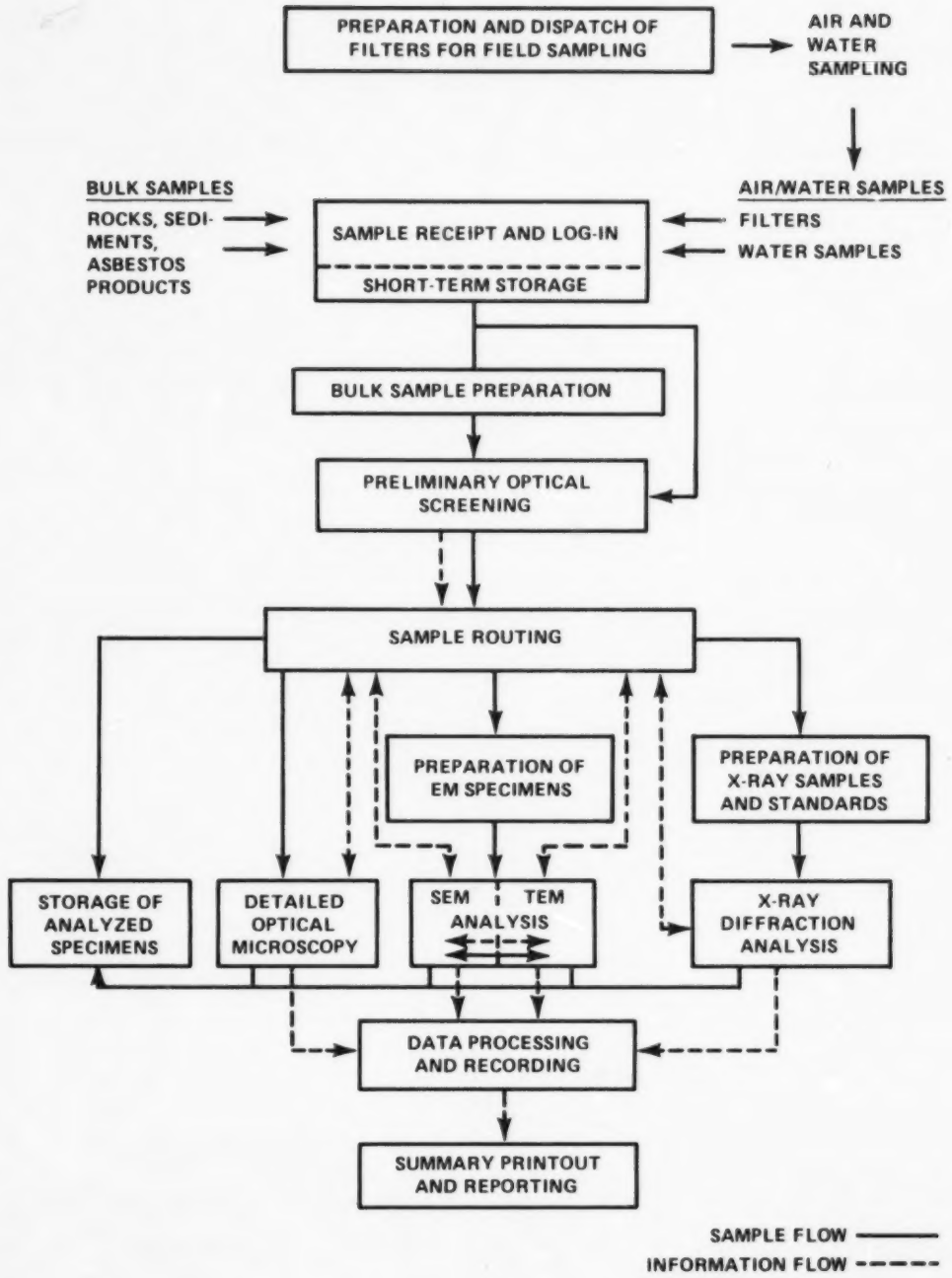

Figure 1. Sample flow sheet and work stations.

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All samples are to be received, unpacked, and logged in a designated area which should be provided with a closed refuse-disposal unit for discarding packing materials. Immediately after unpacking, each sample should be assigned an identification code and entered into the computer-based data log along with information about its origin, classification, and quantity.

After these procedures are complete, the samples will be transferred into the optical screening area where in-coming bulk samples of insulation, plant and road dusts, or any other possible asbestos-containing material submitted for analysis should be examined for the obvious presence of asbestiform substances and possible interfering material or fibers such as cellulose and other organics, glass wool, gypsum, perlite, vermiculite, and quartz. The uniformity of the sample must be checked and the desirability of separation or dispersion of large clumps established. Filters from air or water samples must also be checked upon receipt for the presence of the extraneous material listed above as well as the extent and uniformity of loading.

This optical screening can also serve two additional purposes: (1) to form the basis for selecting initial sample preparation and analysis methods and (2) to identify major sample constituents to the extent possible.

Following the recommendation made at the time of screening, which is to be logged into the data file, the sample is routed through appropriate preparation and analysis procedures as illustrated in the flow sheet and discussed below in the corresponding order.

2.3 Optical Microscopy

Techniques and instrumentation for optical characterization of particulates are well-established. Counting of asbestos fibers can be accomplished by phase-contrast microscopy on a suitable filter(11). The filter is immersed in a liquid to make it transparent and the phase-contrast optics renders clearly visible any fiber in the filter with a refractive index different from that of the liquid.

Using a research-grade binocular polarizing microscope equipped with analyzer and polarizer units, a knowledgeable petrographer can identify the mineral type of any fiber within the size range of optical microscopy. Suitable immersion oils that nearly match the refractive index of the fibers are used to

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"identify" the minerology of the fibers. "Dispersion" staining by using a central or annular stop and immersion in a refractive-index liquid that nearly matches that of the fiber produces colored fiber images due to different dispersion curves of the liquid immersion media and fiber(12). The colors produced are characteristic of each asbestos mineralogy.

On occasion it may be necessary to examine petrographic thin sections or grain amounts of ground rocks to establish the relative occurrence and habit of the relevant mineral types. (It is recommended that preparations of thin sections be handled through laboratories which specialize in this service at modest cost.)

Precise measurements of nonfibrous particle dimensions in the optical range (>1 μm in diameter) can be accomplished by using an image-shearing feature somewhat similar to the focusing aid on some expensive 35-mm cameras. The shear control on the microscope can be coupled to an electronic digital readout of the displacement of the two images displayed. Histograms of particle sizes can be obtained readily.

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2.4 X-Ray Diffraction

Inasmuch as the emphasis in the proposed particulates analysis laboratory is to be the characterization of individual microscopic particles, bulk methods such as x-ray diffraction are not directly applicable. Nevertheless x-ray diffraction is a valuable supplement to light and electron optical analytical techniques(13). In many cases, the technique can serve to identify the main crystalline components of the sample and ensure that fragments of these constituents are not misidentified as asbestos fibers.

2.5 Electron Microscopy

In all critical cases, the identification and verification of asbestos or other particle concentrations will depend on electron microscope observations. Two basic types of electron microscopes are available for this work, and because their individual capabilities are somewhat distinct, but complementary, any state-of-the-art laboratory must utilize both(14)(15).

Scanning electron microscopy. The conventional (SEM) utilizes relatively low voltage (15-30 kV) to produce modest resolution (10-50 nm) images with backscattered or secondary

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electrons. Opaque specimens are mounted on a specimen stub and coated with a thin film metal or carbon to eliminate charging. (Samples imaged by transmitted electrons require the same procedures as transmission electron microscope (TEM) samples, as discussed below.)

X-ray emission spectra, induced by the relatively high intensity electron beam, are used to obtain a qualitative measure of the elemental composition ($z > 10$) by comparison with standards, which provides a means of distinguishing between minerals which differ sufficiently in composition. The concentration of particles in air or water samples is determined by visually counting the number meeting pre-established criteria (length, aspect ratio, shape, composition, etc.) that are observed per unit area in the SEM and applying scale factors for air or water volume, dilution factors, and SEM magnification. This is a tedious, time consuming procedure subject to serious subjective (operator) errors. Furthermore, the resolution is limited to 100 Å at best so that accurate size measurements or counts are not yet possible for single fibrils of chrysotile, for example. Automatic image analysis (AIA) techniques are now available to rapidly determine size, aspect ratio, and probable identification of particles with dimensions greater than 0.1 x 1.0 μm as discussed in Section 2.8.

Transmission electron microscope. These instruments utilize relatively high voltage (80-200 kV usually and sometimes to 1000+ kV) to produce high resolution (to 0.1 nm in optimum cases) images with transmitted electrons. These TEM instruments can be adjusted to produce electron diffraction patterns from individual particles to permit determination of crystal unit cell parameters and hence positive crystal phase identification by knowledgeable microscopists as discussed below.

Some TEM models, primarily designed for analytical microscopy, are equipped with beam scanning controls (STEM/TEM)* and a nondispersive x-ray spectrometer for elemental analysis for qualitative particle analysis and identification as with the SEM. Specimen-preparation procedures for the TEM or STEM are more complicated than SEM preparations and much more time is required to examine a specimen to locate particles and orient them for obtaining optimum diffraction data and imaging.

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* This terminology refers to a TEM fitted with scan coils and a lens capable of producing a fine probe (∿50 Å) at the specimen. The term TEM/SCAN is sometimes used for such instruments.

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Electron diffraction analysis. In cases where it is important to obtain a positive identification of an asbestos variety, it is necessary to use electron diffraction to determine the crystal structure of individual particles. The TEM employed for this work must be equipped with a good goniometer stage to tilt each particle in turn so that a major crystallographic direction is parallel to the electron beam (a "zone axis" orientation).

Detailed descriptions of the identification of microcrystals from single-crystal diffraction patterns are available in texts and the research literature. In brief, interpretation of SAD patterns is accomplished by measuring two primary d-spacings (largest d-spacing or smallest reciprocal lattice vectors) on the pattern and the angle between them. The measured spacings are compared by computer with calculated inter-planar spacings and angles from preselected known structures. Accurate calibration of the camera constant is essential, and this is usually achieved by vacuum coating the sample with a thin film of gold so that a standard pattern is superimposed on the SAD image.

A computer logic program, designated SEARCH, was developed at USS Research to expedite comparison of the SAD data with a large number of reference compounds. It includes the effects of lattice symmetry.

2.6 Electron Microscope Sample Preparation

The major problem in electron microscope sample preparation for particulate analysis is obtaining properly dispersed loadings and eliminating contamination. For either SEM or STEM the desired dispersion corresponds to about 5 $\mu g/cm^2$. This loading minimizes interference in the x-ray collection (due to particles being too closely spaced) without excessive searching, yet permits a sufficient number of particles to be located so that a reasonable statistical confidence level is achieved, as discussed in a following section.

As part of this contract, we conducted a survey of laboratories performing asbestos analysis. Currently, the preferred method of sample preparation for EM analysis is the modified Jaffee wick(16). Other methods such as micropipeting or condensation washing have been abandoned because of their difficulty. In the Jaffe method, particles are collected on a porous filter medium, which is carbon-coated and dissolved, leaving the particles enmeshed in the carbon film. However, the carbon film is a replica of the filter surface, and therefore, there is a

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strong background texture in the substrate with the Jaffe wick technique. This is not the ideal preparation method for AIA as the texture of the carbon film interferes with the computer-controlled analysis. One of the most important advances in sample preparation that could be made would be to develop a background-free substrate for performing automated asbestos analysis, as discussed in Section 7.

2.7 Counting Statistics

At the optimum level of loading, which must be low to minimize interference, a serious difficulty arises from contamination because of the statistical weight given to single observations. It has been reported by Cook(17) that blank analyses from some ashed filters may show from 1 to 5 fibers per grid opening of a 200 mesh grid. Care must be taken that the observed fiber count is differentiated from the background count. Contamination from extraneous sources may be introduced at any point from sample collection to final preparation procedures. However, the clean facilities provided in the proposed laboratory would eliminate gross contamination during the specimen preparation stage. Hence, contamination of the filter media used to collect the samples would be a likely problem source(17).

2.8 Automated Electron Microscopy

Background. Traditionally, the concentration and size distribution of asbestos fiber or other particulate species of interest in environmental samples have been measured with optical or electron microscopes by manual counting, sizing, and identification procedures. There are several intrinsic limitations to manual techniques:

1. Only the species with characteristic shapes can be counted and identified.

2. The analysis is time-consuming and costly.

3. The operator must locate and identify the feature. Thus, subjectivity is unavoidable and operator fatigue is a problem.

4. The absolute number of particles or fibers counted is necessarily small, leading to large uncertainties because of the statistics of small numbers.

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Microscopists have long been aware of the problem and interested in developing automated techniques. Although automated optical microscopes have been available for some time, the physical size of asbestos fibers and the complexity of environmental samples have limited their use for particulate analysis.

A major breakthrough in the analysis of particulate samples occurred with the development of the LeMont digital beam-control system for electron microscopes. Originally the instrument was generally regarded as having limited value because it provided a limited, inflexible capability for x-ray analysis and classification of particles. However, after USS Research interfaced the LeMont system with a software-based EDS system, a new dimension of flexibility, pattern-recognition capability, and speed was realized(18). Since that innovation, LeMont, Ontario Research Foundation, and Tracor Northern have introduced systems interfaced with energy dispersed spectrometers for x-ray analysis.

Function of Components. Automated electron microscopes have three basic components: the electron microscope, the beam-control system, and the x-ray microanalyzer. Functionally, the SEM or STEM is used to produce an image via the interaction of the focussed electron beam with the sample. The advantages of the SEM/STEM—high resolution, large depth of field, variable magnification, and scanned electron beam—make them well-suited to remote computer control and real-time point-by-point analysis of the signal.

The digital beam-control system (DBCS) is the second component of an automated electron microscope. This system converts the normal analog scan in the SEM/STEM to a digital motion. The DBCS then samples the signal produced by the microscope at each point to determine whether a particle has been located. If not, it directs the beam to a new location. If so, the DBCS directs the beam through a series of steps to measure the dimensions and shape of the feature. Finally, it repositions the beam on the particle, initiates the x-ray analysis, and stores the physical size/shape parameter on magnetic disk.

Characteristic x-rays are generated whenever an electron beam interacts with a material in the electron microscope. A well-developed technique for collecting and analyzing the x-ray spectra then generated is termed energy dispersive spectroscopy (EDS). Characteristic x-rays produced by all elements with atomic No. $\geq$11 (sodium) are detected simultaneously. Generally, there are significant variations in

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sampling conditions which make quantitative analysis of the x-ray spectrum difficult. However, although the count rate may vary, the relative intensities of the characteristic x-ray peaks from a specific compound are highly reproducible. Therefore, the knowledgable user quickly learns to identify compounds on the basis of the position and relative intensities of the x-ray peaks; that is by pattern recognition.

Such capability is readily programmed in all but the most subtle situations, and when combined with the size/shape data from the beam control system, amounts to a quantitative characterization of individual species.

Current Capabilities and Limitations of Automated Electron Microscopes. Several automated SEM's and STEM's are operational, with the number of instruments growing rapidly. The automated electron microscope permits characterization of the size/shape/composition distribution of all particles in a sample for complete microscopic sample characterization. Presently, hardware/software combinations are available for the measurement and analysis of individual particles in times of 1 to 2 sec per particle. The size of the particle can vary over several orders of magnitude, and the shape can vary from equiaxed to fibrous, as illustrated in Figure 2, which shows the conventional image and the digital measurement pattern. The lower limits on the analysis of asbestos fibers in conventional SEM are fibers $\leq$0.1 um wide and $\leq$1 um long. Thus, in the SEM, we measure asbestos fibers $\leq$0.1 um x 1.0 um manually and measure the dimensions of larger fibers automatically. We expect that implementation of our automated procedures on STEM's with higher brightness sources will make it possible to extend the size distribution down to a lower limit of 10 nm. The conventional and digital images of a typical chrysotile fiber are shown in Figure 3.

Another basic limitation of automated electron microscopy is sample-preparation methodology. The human eye easily picks out single asbestos fibers on a complex background even when they are partially obscured by other material. This is a nontrivial problem for the automated electron microscope. Programs have been developed to handle overlapping or partially obscured fibers, but the occurance of pronounced granularity (as in Figure 2) in the background limits the accuracy considerably. Ideally, every sample should have a uniform featureless substate, as in Figure 3. If this is not the case, a curved fiber as in Figure 3 cannot be measured, and a significant amount of analyzer time can be spent analyzing background instead of real particles.

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a) SCANNING TRANSMISSION

b) DIGITAL

Figure 2. Illustration of digital measurement in STEM made on the SEM. (a) STEM micrograph, (b) Digital measurement of equiaxed and fibrous particles. Note the texture of the background in (a) which interferes with the automated measurement of single fibrils.

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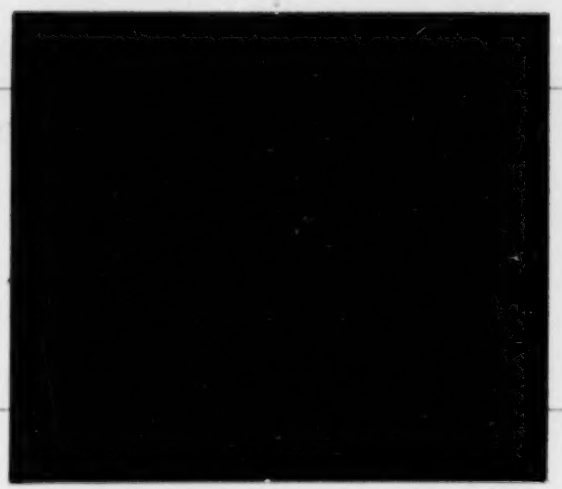
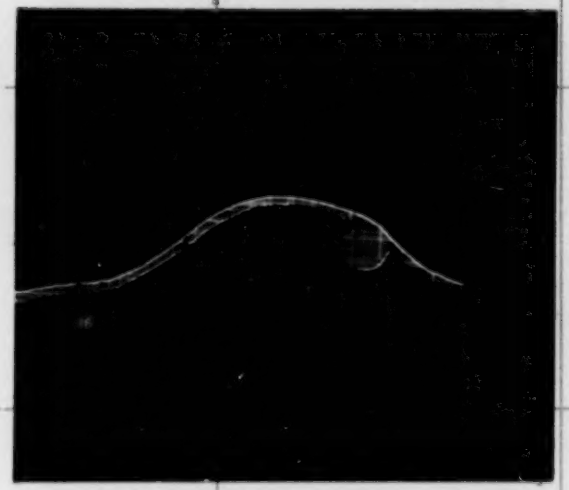


a) STEM Image 10,000X



b) Digital Measurement 10,000X

Figure 3. Illustration of fiber measurement in STEM made on SEM. (a) STEM micrograph of fiber, (b) digital measurement of fiber using LeMont Scientific "grid" program. The program resolved the fiber and circular particle adjacent to it. Sample preparation method used was condensation washing. Sample supplied by W. C. McCrone Associates.
Note the featureless background in (a).

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In summary, the current advantages of automated electron microscopy are

1. Rapid, reproducible characterization of the size/shape and composition of particles and fibers.

2. Reproducible measurement of size and shape, thus eliminating subjective decisions on whether or not to count a fiber.

3. Improved statistics from counting larger numbers of fibers and larger areas.

4. Complete sample characterization rather than just the species thought to be of interest.

8-1/2"

The current disadvantages are

1. Sample-preparation procedures capable of providing featureless substrates are not well developed.

9-1/8"

2. Partially obscured fibers are not readily measured.

2.9 Proposed Best Available Analysis Technology

Even experienced microscopists find electron-diffraction identification of unknown minerals to be very time consuming. Unfortunately, for definitive analysis of amphibole asbestos the use of electron diffraction is essential to corroborate the chemistry obtained by x-ray spectroscopy. The correspondingly high cost of positive particle identification by electron diffraction is especially serious with water or ambient air samples where a wide variety of particle types can occur. In some cases an acceptable compromise is to classify a large number of particles according to type classes on the basis of x-ray spectroscopy and aspect ratio by automated or manual electron microscopy as discussed above. Representative particles in type classes are then positively identified by the electron-diffraction procedures outlined above. Since every particle is identified, this method introduces a statistical uncertainty into particulate analysis. However, assignment of a probable error to the apparent concentration of critical constituents, such as amphiboles, could be based on the relative number of "identified" and "classified" particles.

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This procedure may be termed the currently "best available analysis technology" as summarized in Table I.

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TABLE I. BEST AVAILABLE TECHNOLOGY PARTICULATE COUNTING

Record size, shape, composition and location of all particles.

Assign each particle to type category.

Record, measure, and interpret SAD patterns from randomly selected particles.

Estimate uncertainty in the identification of each particle type. Sources of uncertainty:

6-1/2"

a) Compositional interferences.
b) Crystallographic interferences.
c) Pattern quality.

9-1/8"

Continue analysis until predetermined statistical criteria are satisfied.

a) Variability in number per unit area.
b) Confidence level of concentration.

For uncontested analysis, this counting and identification procedure ensures the production of reliable and reproducible data. But, if verification of the size, aspect ratio, or identification of any one or all particles should be required for any specific reason, the availability of retrievable stored information will greatly facilitate the process.

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SECTION 3

BUILDING AND CLEAN ROOM

3.1 Introduction

The general design of a building (Figure 4) to house the proposed particulates analysis facilities is based on the following general premises. It is to be a free-standing, one-story structure large enough to house an optimal staff and all essential requirements, including self-contained sample-receiving area, clean rooms, laboratory space, staff and administrative offices, air-handling equipment, computer room, conference room, library, and canteen. This structure must be free-standing to afford protection from the contamination that will inevitably result from the air-handling system should the facility be incorporated into or contiguous to a larger structure. Obviously, the local institutional setting will affect some of these choices. The nominal dimensions of the proposed building, which includes all the above-mentioned features, are 56 feet by 104 feet for a total area of about 5800 square feet. Approximately 1/4 of the space is devoted to clean rooms, 1/4 to laboratories, 1/4 to offices, and 1/4 to utilities.

The philosophy underlying the design of the building is that the clean areas, laboratory areas, and office areas are spacially separated. The air-handling equipment for the building can thus be set up so that pressure differentials are maintained between the clean areas, the laboratories, and the office space. This will permit smoking and other activities in the office space without affecting the laboratory and clean areas.

3.2 The Office Space

The office area shown in Figure 4 is isolated from the clean areas in the building by spring-loaded swinging doors in the central corridor, which are normally closed. The air to this area is not specially filtered or cleaned in any extraordinary way. The rooms in this area are separated by movable

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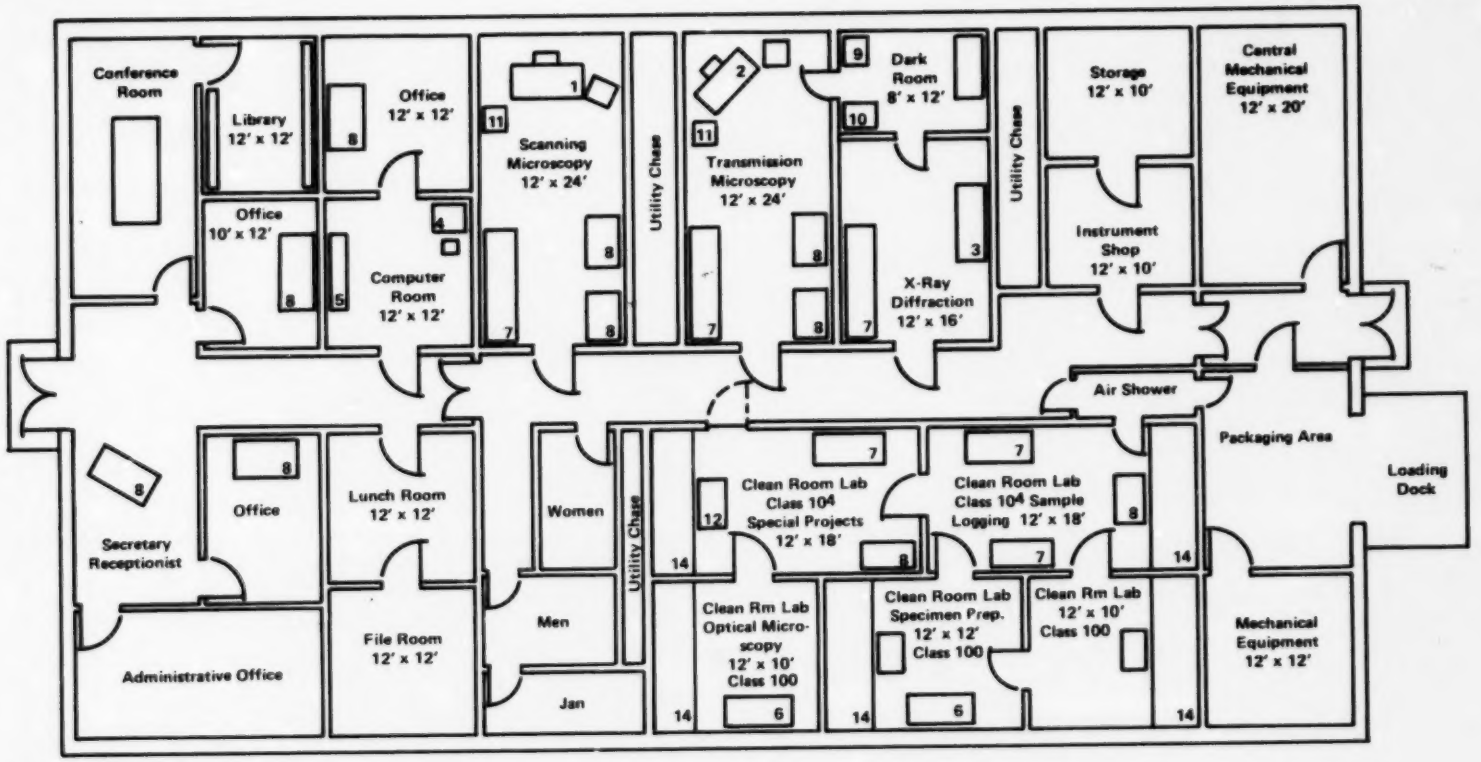
Conference Room
Library 12' x 12'
Office 12' x 12'
Office 10' x 12'
Computer Room 12' x 12'
Scanning Microscopy 12' x 24'
Utility Chase
Transmission Microscopy 12' x 24'
Dark Room 8' x 12'
X-Ray Diffraction 12' x 16'
Storage 12' x 10'
Central Mechanical Equipment 12' x 20'
Instrument Shop 12' x 10'
Air Shower
Packaging Area
Loading Dock
Secretary Receptionist
Office
Lunch Room 12' x 12'
Women
Men
Jan
File Room 12' x 12'
Administrative Office
Clean Room Lab Class 10⁴ Special Projects 12' x 18'
Clean Room Lab Class 10⁴ Sample Logging 12' x 18'
Clean Rm Lab Optical Microscopy 12' x 10' Class 100
Clean Room Lab Specimen Prep. 12' x 12' Class 100
Clean Rm Lab 12' x 10' Class 100
Mechanical Equipment 12' x 12'

EQUIPMENT LEGEND:

1. Scanning Electron Microscope
2. Scanning Transmission Microscope
3. Automatic X-Ray Diffractometer
4. Computer Terminal
5. Disk Storage
6. Optical Microscope
7. Work Benches
8. Desks
9. "Logetronics" Printer
10. Enlarger
11. Image Analysis Equipment
12. Evaporator
13. Class 100 Fume Hoods
14. Circulating Fan & Pre-Filter Enclosure (Typ. Five Places)

FLOOR PLAN 1

Figure 4. Plan of proposed laboratory showing office, laboratory, and clean room areas. Placement of the essential instruments is also indicated.

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partitions so that as staffing levels change the accommodations can be altered.

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The lunch room with vending machines is an optional nicety that may be deleted if other facilities are available locally. However, the computer-terminal room and the small library are essential to the operation of a good particulates laboratory. The computer terminal can be used to retrieve the results of analyses in a form ready for reporting or to locate other information such as the scheduling of samples for analysis. The library should contain a small selection of journals and a good collection of reference books on mineralogy, crystallography, geology, and federal and state government air and water quality regulations.

3.3 The Instrument Area

The air supplied to the instrument area should be filtered with 99.97 percent HEPA filters and kept at slightly positive pressure relative to ambient. A major consideration in this area should be the floor construction in the microscope rooms. The floor must be specially reinforced so that there is no appreciable ambient vibration (less than 1 μm amplitude) and also properly supported so that movement of people about the room will not deflect the beam of the microscope. It is also essential to reduce alternating electromagnetic fields to very low levels, which can be accomplished by assuring that the air-handling fans and motors are positioned a considerable distance away from this area. Ballasts for fluorescent lighting are best placed in a remote position. Because the mechanical vacuum pumps of electron microscopes emit oil vapor droplets and both electromagnetic and mechanical vibration, these should be located in the narrow utilities corridors adjacent to the laboratory space. Each laboratory should be supplied with recirculating chilled water (containing corrosion inhibitors) for the cooling of the instruments.

3.4 Clean-Room Area

Several visits were made to facilities performing asbestos analysis. Our survey of these indicates that no one (including USS Research) is maintaining proper clean-room procedures and standards. In most cases the asbestos-analysis activity developed after the establishment of the laboratory. Thus, clean rooms were accommodated to the existing facilities, not vice versa. However, even with a well-designed clean room,

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it is essential to enforce a quality assurance program among all personnel operating the facility. We identified several major problems in the operation of clean rooms:

1. Poor clean-room practice.

2. Lack of independent air-filtration systems for each clean-room area.

3. Lack of facilities, carbon evaporators, etc., necessary to complete preparation of samples without transporting them to other areas of the building.

4. Lack of adequate sample storage and handling facilities within clean rooms.

5. Lack of a systematic monitoring program for possible contamination of clean-room air, utensils, and supplies.

6. Inadequate hoods for handling the evaporation of solvents.

Clean room modules can be purchased from a number of suppliers and several types are described below. All the proposed systems rely on a vertical laminar flow of air rather than the less effective horizontal flow. For this application, a floor grating required for full vertical laminar flow has been rejected in favor of floor-level return units. Three different philosophies are presented below with comments on the advantages and disadvantages of each system. However, none of the designs can supply the most important ingredient of a clean room - the human element and "thinking clean."

Option A, Weber Technical Products. An example of a typical modified vertical-flow clean room manufactured by Weber Technical Products is shown on the left of Figure 5. The makeup air to this room is prefiltered and moves vertically from the ceiling through a 99.97 percent HEPA filter bed at a velocity of 100 ft/min. Solvent fumes are recovered from the air in some of the clean rooms by using larger than normal amounts of makeup air and using activated graphite filters on the return air. With this design some care must be taken not to introduce dirt into the room from people and packaging.

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BUILDING ROOF LINE
BOTTOM/ROOF JOISTS
PRE-FILTERED MAKE-UP A12
CENTER SUSPENSION MODULES
SIDE SUS-PENSION MODULE
FLOOR
5'-0"
8'-0" CEILING HEIGHT
PRE-FILTERED MAKE-UP A12
HEPA FILTERS, SERVICED FROM WITHIN ROOM
PRE-FILTER INCLUDING GASEOUS & ODOR FILTRATION
FLOOR
MODEL 20 CHANNEL HOODS BY FILTECH INC.
OR
FILTER-FLO CEILING DIFFUSER BY WEBER TECHNICAL PRODUCTS
CLEAN AIR CEILING MODULES
BY
ENVIRONMENTAL AIR CONTROL, INC.
NOTE: CLEAN ROOM SUPPLIER TO DESIGN, INSTALL, TEST AND OBTAIN CERTIFICATION OF CONFORMANCE TO SPECIFICATIONS.
TYPICAL SECTIONS OF CLEAN ROOM SYSTEMS AVAILABLE FROM INDICATED MANUFACTURERS CLASS 10^2 AND 10^4 AIR

Figure 5. On the left is a cross-section drawing of a typical vertical flow clean room designed by Weber Technical Products. On the right is a view of the clean work station concept provided by Environmental Air Control.

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Option B, Environmental Air Control Inc.. An alternative method to cleaning all the room air is available from Environmental Air Control, Inc. This design, shown in Figure 5 on the right, has suspended air-filtering modules above the work surfaces only. This has the advantage that the work area can be maintained clean without special precautions being taken to reduce dirt being carried into the room. For those rooms where solvent vapors are a problem the side-suspension module, which exhausts most of the vapors through a side vent, would be the preferable geometry. As in the previous design the solvent vapors can be diluted and removed by the addition of larger amounts of makeup air and activated graphite filters.

Option C, Class 100 Fume Hood Contamination Control Incorporated. By far the simplest method for obtaining Class 100 conditions in a limited work area is to use a fume hood designed to meet Class 100 specifications. This eliminates the necessity of purchasing expensive clean rooms, air showers, and other air-handling equipment shown in Figure 4. If Option C is selected then a modified laboratory floor plan, as shown in Figure 6, would be required. This configuration eliminates the mechanical equipment room required for the clean room and is now conditioned and heated by the regular building system. The only special requirements for this configuration are four exhaust vents in the ceiling for the fume hoods and a slightly positive ambient pressure similar to that in the instrument area. The fume hood made by Contamination Control, Inc. (Model 932) is ideal for this purpose. This hood is particularly flexible in that it can be set for 50 or 100 percent exhaust, depending on whether harmful vapors are present or not.

It is our belief that the last option will be perfectly adequate for the requirements of this laboratory. The cost of this system is about one-tenth that of the conventional clean room. If stringent control is exercised over the operation of the general area, we know of no reason why it could not be adopted. Four hoods, each 6 feet wide, should be sufficient for this laboratory. The costs for options A and B are approximately $120,000 whereas four hoods would require an expenditure of $14,000.

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Figure 6. Modified floor plan of the "clean area" if the Contamination Control, Inc. fume hood is adopted.

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SECTION 4

ANALYTICAL INSTRUMENTS AND DATA RECORDING AND PROCESSING EQUIPMENT

4.1 Introduction

To fulfill the multiple functions of the particulates analysis laboratory, that is to keep abreast of and contribute to advances in particulate analysis methodology, to analyze a wide variety of air and water samples, and to corroborate, in crucial cases, data obtained by others, the laboratory must be equipped with top quality light and scanning and transmission electron microscopes and an x ray diffractometer. A multi-terminal computer system is also required to expedite monitoring of the status of the analysis of current samples and retrieval of earlier data needed for comparison in reporting programs. The criteria for selection of particular manufacturers and models include specifications, performance, actual compatibility with expected future developments, availability of replacement parts, service, and relative cost.

Another factor crucial to effective utilization of the equipment is user acceptance of the instruments selected for the particulates analysis laboratory. In particular, the electron microscopists hired to staff the laboratory may have had previous favorable or unfavorable experience with a particular manufacturer and may be unhappy with our choice of microscopes. To avoid such a reaction, it is important that the users understand and accept the basis for selection of particular microscopes.

Specific recommendations for the various items required for the laboratory are based on our own extensive experience, visits to manufacturers' and users' laboratories, and contacts with knowledgeable people in the field. A discussion follows on the types of instruments required and at the end of this section our recommended manufacturer, model numbers, and costs are given. A brief discussion of the reasons for our selection is presented.

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4.2 Optical Microscopes

High-quality optical microscopes of various types are essential to the laboratory as every sample must be screened by low-power microscopy on receipt; in many cases the analysis may be accomplished by phase contrast on polarized-light microscopy alone. In addition, each work station must be equipped with a low-power stereoscopic microscope with zoom magnification to facilitate such sample preparations as mounting for electron microscopes. Accurate size measurements of large particles can be accomplished with an image-shearing microscope. Recommended manufacturers and model numbers are listed below.

Stereo Microscope

Bausch & Lomb Stereoform — $900.00 each
Model BVB-73 with Nicholas Illuminator — (5 required)
Reason for this selection: Standard microscope readily available from various laboratory supply houses.

Polarizing Light Microscope

1st Choice: Olympus BHP-100A, trinocular automatic camera, polarizing phase contrast optics. — $9200.00 each (2 required)
Reason for this selection: There are many high-quality polarizing microscopes but the model chosen will do the same job at a lesser cost.

Image-Shearing Microscope

1st Choice: Vickers CCP — $9700.00
Reason for this selection: This microscope is unique.

4.3 X-Ray Diffractometers

The major crystalline phases in powder samples can be routinely identified by x ray diffraction. Complete characterization of bulk samples as well as heavily loaded filters may be required in many instances and will also help to prevent misidentification of fragments of major constituents as asbestos. New techniques for identifying asbestos particles by x ray diffraction are under development and the capability of inducting such methods as they evolve should be available in the particulates laboratory.

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First Choice

Siemiens Automatic D-500 X-Ray Diffraction System ~$120,000

Reason for this selection: The Siemiens system offers automatic specimen changing and sample rotation which are not available in other units. Software to analyse quantitatively for a variety of minerals is also available with this system.

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4.4 Scanning Electron Microscope (SEM)

Generally, SEM manufacturers are highly competitive in terms of instrument capability; resolution, accelerating voltage, magnification range, signal detectors, image processing, and accessory items are comparable. Because of this, only American and Canadian manufacturers are considered as potential sources of SEM's. Of these three manufacturers, AMR, Inc. and SEMCO, Inc. were visited to evaluate performance, ease of operation and maintenance, and potential for future development in the area of image analysis. ETEC, Inc. was not visited because of our personal familiarity with the instrument (USS has five ETEC's); therefore AMR and SEMCO are compared with ETEC. Our comparison of the instruments is summarized in Table II.

TABLE II. COMPARISON OF SEMCO AND AMR WITH ETEC

	AMR	SEMCO	ETEC
Ease of Operation	Good	Good	Excellent
LaB_6 Gun Performance (Demo)	Excellent	Poor	NA
Service	Mixed G&P	NA	Mixed G&P
Remote Computer Control of Magnification	Will provide	Will provide	
Automated Stage	NA (will provide)	NA	Excellent (costly)

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TABLE II (CONTINUED)

	AMR	SEMCO	ETEC
Backscattered Electron Detector	Good	Excellent	Good
Transmitted Electron Detector	Excellent	Good	Poor
Transmission Holder	Poor	Poor	Poor
Chamber Access	Good	Excellent	Good
Mechanical Design of Stage	Excellent	Excellent	Good
Future Developments	Microprocessor control of SEM(uncertain)	Microprocessor control of SEM(in 1980)	Microprocessor control of SEM; improved lens design (uncertain)
Zoom Mag.	Good	Good	Excellent
LaB_6 Lifetime	300 1000 hr	100 300 hr	NA

Specific Comments. LaB_6 sources are just becoming available. These cathodes are considerably more difficult to align and maintain than tungsten filaments; they may not even be suitable for multi-user laboratory environments, but their advantages dictate that any instrument be equipped to use LaB_6 emitters.

AMR's high-vacuum system is very massive, has metal ring seals, and would provide the best operating environment for a LaB_6 system. It has clear advantages over both ETEC and SEMCO.

AMR has only an 8 bit addressable digital scan generator (DSA), a marked disadvantage when compared with ETEC and SEMCO, both of which have 12 bit DSA's.

The access to SEMCO's specimen chamber is better than that of ETEC or AMR.

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SEMCO has a better backscattered electrons (BSE) detector than ETEC, AMR, or USS Research, and an internal TV scan capability. This is very useful for screening of large sample areas.

AMR has a better STEM detector than SEMCO or ETEC; it is comparable to the one we have developed at USS Research.

Neither SEMCO or AMR have as flexible zoom magnification units as ETEC—this is an important user concern.

AMR has the most flexible character display for micrograph labeling.

SEMCO will have a complete microprocessor control of lens current, magnification, and accelerating voltage SEM in production during 1980. AMR and ETEC are in a similar development program, but lag SEMCO by at least a year. This capability is an important long-term factor in terms of AIA.

SEMCO is a new manufacturer, with a significant cost advantage over ETEC and AMR, but with no long-term proven reliability index. Both ETEC and AMR rate high here.

ETEC and AMR both have highly mixed reviews in terms of field service. Small remote users tend to be ignored, but more importantly, the competence of field service engineers varies dramatically.

Comparable scanning transmission electron micrographs are presented in Figure 7 for ETEC and AMR. The benefit of the increased gun brightness due to LaB_6 filaments is most noticeable in Figure 7 taken at 30,000X on the AMR. Using the tungsten filament on the ETEC, we could not go above 10,000X.

Summary. Evaluation and comparison of SEMCO and AMR with ETEC suggests that important changes in design will take place within the next 18 months, and therefore the following observations are subject to rapid change in the near future. Although all three have excellent instruments, the recommended choice would be

1. AMR—if their single-crystal LaB_6 performance is maintained in the field for 18 months. Estimated cost is $110,000.

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a) STEM Tungsten 10,000X

b) STEM LaB_6 30,000X

Figure 7. STEM micrographs recorded on ETEC SEM using a tungsten filament (a), and AMR SEM using a LaB_6 filament (b).

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2. ETEC—if their high-resolution lens becomes a reality. Estimated cost is $100,000.

3. SEMCO—if their microprocessor-controlled SEM becomes available, and if they develop a functional single-crystal LaB_6 cathode. Estimated cost is $85,000.

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In each case, the gun area should be ion-pumped and isolated from the chamber, and the manufacturers should be required to provide necessary interfacing to an automatic image analysis system.

A specification sheet required for ordering this instrument is included as Appendix A.

4.5 Scanning Transmission Electron Microscope

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The major difference that separates the STEM's on the market today is that some use microprocessors to control the operation of the system. These instruments are generally simpler to use and are therefore preferred. The two microprocessor-controlled STEM's are the Philips 400 and the Hitachi 600. We also included in our evaluation the JEOL 100CX unit because the manufacturer has an excellent reputation. The specifications of other instruments were examined and because of inadequacies in their capabilities no further tests were run on these instruments.

Visits were paid to the JEOL and Hitachi Customer Service Laboratories and to Oak Ridge National Laboratory where a Phillips 400 is in use to conduct evaluation tests. Representative specimens of serpentine and amphibole asbestos were used as test specimens. Particular attention was paid to the ease of specimen loading and of switching between the various modes of operations in STEM, TEM, SEM, SAD, and x ray microanalysis. Other factors compared included the x ray count rate, and signal-to-noise ratio on standard samples, as well as the rapidity of recording micrographs, inserting identifying labels, and obtaining computer printouts of x ray and other data. These factors are somewhat dependent on operator experience and skill, and in this respect the demonstration at JEOL was superb. Here an impressive amount of micrographs and data was acquired in a relatively short time.

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Table III summarizes some important characteristics of STEM's.

TABLE III. COMPARISON OF STEM's

	Philip 400	Hitachi 600	JEOL 100CX
Ease of Operation	Good	Excellent	Good
Microprocessor Controlled	Yes	Yes	No
Takeoff angle Solid and X-Ray Detector	Poor	Excellent	Poor
Magnification Range	Good	Excellent	Good
Contamination Rate	Excellent	Good	Good
Digital Data Logging on Film	Yes	Yes	Yes
Operation Modes-Switch Selectable	Yes	Yes	Yes
Specimen Goniometer Stage	Good	Good	Good
Cost	Expensive	Moderate	Moderate

For the analytical purposes of the proposed laboratory we recommend that the order of performance is Hitachi 600, JEOL 100CX, and Philips 400. The Philips 400 is a very good analytical tool but its extra cost is not warranted for this application. The JEOL 100X was downgraded in our evaluation because it lacked a microprocessor to control operation. The reasons for preferring the Hitachi 600 are given below.

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First Choice; Hitachi HU-600 2 $310,000
Reasons for selection over other makes and models: This microscope has the highest takeoff angle for x rays and the lowest magnification when used in the STEM mode, which are essential factors for automatic image or x ray analysis of particulates. Additionally, the microprocessor control of this microscope makes for simple operation and its relatively low price means that it is cost-effective.

Second Choice: JEOL-100CX

Third Choice: Philips 400

4.6 Digital Beam Control System

To realize the advantages of automatic image analysis for particulate characterization, as discussed above, both the SEM and STEM must be fitted with Digital Beam Scan Control Systems.

Two manufacturers supply equipment specifically designed and developed to be interfaced with electron beam instruments: Tracor Northern and LeMont Scientific. Of the two, LeMont has the best software for automated measurement of particle size and shape. In fact, Tracor Northern does not yet have a program to characterize fibers.

We have two LeMont systems, both of which have been extremely reliable and easy to use. An informal survey of other users indicates this is generally the case; however, users have mixed experiences in terms of field service when failures occur. The recommended Digitial Beam Control System is LeMont Scientific B-10, $60,000.

Reason for this choice. Availability of software to perform fiber analysis. Compatibility with all existing energy dispersive x ray analyzers.

4.7 Energy Dispersive Spectrometer

To characterize size/shape/and composition, the Digital Beam Control System described above must be interfaced to a Energy Dispersive Spectrometer (EDS). U. S. Steel interfaced a LeMont System to a Tracor Northern Analyzer some years ago and has developed an extensive library of programs for

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characterizing and classifying particles according to size/shape/and composition.

Recently LeMont has taken the same approach and developed software to sort x ray data received from an EDS. Their system can be interfaced to any of the current EDS models. Their software permits the user to classify x ray spectra on the basis of relative peak intensity and/or particle size/shape much like the U. S. Steel software.

Several EDS systems are available. Generally they all offer excellent capability to perform quantitative analysis, and massage EDS x ray spectra. We would recommend the Tracor Northern 4000 for the following reasons:

1. Their approach is user-oriented. They permit user modification of software, and offer the capability for user program development which can utilize all the specialized software for spectra manipulation developed by Tracor Northern.

2. Additional x ray spectrometers can be added to the TN-4000 analyzer. This is an important capability. Automated analysis requires speed and precision. Addition of a second spectrometer offers a low-cost means of improving x ray statistics without collecting data for longer periods of time. This capability is unique.

3. U. S. Steel has developed software for sorting a wide variety of minerals on the basis of size/shape/composition. This software has been developed for the Tracor Northern EDS system. Duplication of the software would require an extremely time-consuming effort. In some EDS systems, development of the software is not feasible.

4. Even though LeMont is developing a sorting routine for their Digital Beam Control System, it will not be as flexible as that developed within an environment dedicated to the analysis of x ray spectra, as in an EDS system.

The recommended EDS system is Tracor Northern TN-4000, $30,000.

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4.8 Minicomputer

Extensive use of computer equipment for data logging and processing is strongly recommended. Terminals at convenient locations in the laboratory would be used to log in specimen description and identification upon receipt and to record observations and quantitative results at each point in the analysis flow sheet; hard-copy printouts would be used for reports. The terminals would be used to call up the results to date when starting the next phase of analysis and for management purposes to position the flow of samples through the laboratory. It is to be anticipated that analysis of a number of samples will be in progress at any time, with varying due dates, and it will not be satisfactory to rely on notebooks and informal data records and reports. The instrument recommended is

Ohio Scientific Challenger Data Base Manager System with four remote terminals, rigid disk printers, and custom software.	$22,000

4.9 Miscellaneous Laboratory Equipment

Appendix C is a complete listing of small equipment items required for a laboratory of this type.

4.10 Limitations on Instrument Selection

The equipment recommendations in this report apply only to the unique requirements of a particulates analysis laboratory. A laboratory with a different purpose might select different types and models of instrumentation. Additionally, the service record of all the manufacturers is difficult to evaluate. In the long run the ability of the staff to isolate and repair instrument malfunctions will be the best kind of service.

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SECTION 5

PERSONNEL REQUIREMENTS

The procedures and results of asbestos analysis are subjects of continuing concern and debate. Questions still unanswered have important ramifications for our understanding of the adverse health effects produced by asbestos, the concentration, type, size and shape of fibers required to produce an effect, and finally our ability to develop rational, effective control strategies where they are needed. The particulates analysis laboratory will be expected to make a major contribution to the development of reliable techniques for monitoring particulate concentrations.

The very challenging role to be undertaken by the proposed laboratory demands that it be staffed by scientists and technicians of the highest caliber. Anything less will negate the whole point of establishing a new facility dedicated to excellence in particulate analysis with an emphasis on asbestos. The key individual is the senior scientist selected to inaugurate and direct the facility. Considerable emphasis should be placed on obtaining an experienced person with outstanding credentials.

The specific credentials and technical expertise required for the personnel of the proposed laboratories, and the number of persons required, have been determined from our own experience and those in other laboratories we have studied or visited.

An informal survey among our university colleagues revealed that there is an abundance of both electron microscopists and optical petrographers trained to the masters and doctorate level. However, only a minority of these prospective candidates have the necessary multidisciplinary skills required to perform asbestos analysis or more generally, automated analyses of particulate samples. The other skills required of the transmission electron microscopy candidates are knowledge of mineralogy, crystallography, and diffraction physics. During our conversations we learned of several such people who could perform these tasks adequately and might be available for the laboratory.

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The scanning electron microscopists required should have highly developed expertise in computer-based data collection and manipulation systems. Knowledge of mineralogy and the physics of x ray microanalysis is essential to this position. Candidates with these skills are available.

Optical petrography is the least skilled and the most easily filled position on the scientific staff. We have received many responses showing interest in this type of position.

The last and most important position on the scientific staff is that of the laboratory leader or supervisor. The specific educational background of this person is not as important as established reputation and experience in particulates research. In our opinion, there are very few persons with the necessary qualifications. To attract one of these persons, a full and acknowledged commitment to excellence will be mandatory. This will require adequate initial and ongoing support for developing and maintaining expertise of the highest caliber. We believe that if the laboratory is established as recommended in this report the excellent opportunity presented should be sufficient to attract one of the best individuals in the country.

5.1 Staffing Levels Required for the Laboratory

Recommendations have been developed for two levels of staffing which will be sufficient to run the laboratory at essentially optimum and minimum levels of operation. Priorities for staff additions for intermediate levels are also discussed.

The optimum staff would comprise trained specialists for each type of instrumentation installed in the laboratory, with sufficient support personnel to run all the instrumentation at full capacity. The minimum staff is that required to maintain the instrumentation, stay abreast of developments, and perform a very limited number of special analyses essentially to corroborate results obtained at other laboratories.

For the maximum laboratory compliment we propose the following staff.

4 Scientists	2 electron microscopists 1 petrographer 1 supervisor or senior scientist

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8 Technicians	2 - experienced with electron microscopes 6 - high-school level/ untrained
2 Support People	1 secretary 1 computer operator

Total of 4 professional and 10 support people.

The minimum staff would require the following:

2 Scientists	1 electron microscopist 1 petrographer
4 Technicians	2 - experienced 2 - high-school level/ untrained
1 Support Person	Secretary

Total of 2 professionals and 5 support people.

Ideally, candidates for the professional positions will have advanced training closely akin to particulates analysis and practical experience in the recommended experimental procedures. The minimum level of education and experience level of the scientists is more difficult to define. A bachelors level person might be qualified provided that at least 5 years experience in similar laboratories had demonstrated competence in the work involved. However, our preference is for some postgraduate education (probably at the PhD level) for most, if not all, of the professional staff. The professional staff will be the essential nucleus of this laboratory and it is extremely important that the very best scientists be employed if high-level work is expected.

5.2 Availability and Skills of the Scientific Staff

Currently, laboratories attempting to find qualified persons with experience in the area of asbestos analysis are experiencing considerable difficulty filling these positions. In large part, this is due to the multidisciplinary nature of the analytical skills required. This problem becomes worse when looking for previous experience in automated microscopy because it is a totally new area.

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Thus, we conclude that, with the exception of the senior person, the optimal solution will be to select highly skilled recent graduates, provide them with suitable training courses and adequate lead time to develop expertise in the area of particulates analysis, with an emphasis on asbestos, during the start-up phase of the laboratory. On the basis of our survey, we forsee no difficulty in obtaining qualified but inexperienced personnel.

In Appendix D, we have included the curricula of courses available for certain aspects of particulates analysis.

5.3 Salaries for the Staff

Precise salary scales are not possible in that they depend on the geographical position of the laboratory. However salary ranges can be specified for the laboratory in its maximum staff configuration.

Position	Annual Salary (Thousands)
4 Scientists	1 person ∿ $50 3 persons ∿ $30-$36
8 Technicians	2 persons ∿ $22-26 6 persons ∿ $12-$16
2 Support People	2 persons ∿ $12

In selecting people for the laboratory in its minimum staff configuration, the lower paid scientists, technicians and support people should be dropped, leaving only the most highly skilled component. It is our belief that skimping on the salaries of the top two or three scientists would be false economy.

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SECTION 6

THE LABORATORY GENESIS: TIME SCHEDULE AND COSTS

In the preceding chapters the physical equipment, methodology, and personnel of the laboratory have been described. In this section the time sequence or timetable required to make the laboratory a reality and the costs that will be incurred are reviewed.

6.1 Significant Milestones

Table IV shows a 24-month schedule for the completion of this project. We believe that when the laboratory is approved the distinguished scientist who will eventually run the laboratory should be brought into the project along with the architect. This scientist and architect should work closely together in designing the building in detail, using our conceptual design as a model. There will be adequate time available for the scientist selected to make the necessary visits to laboratories and become fully versed in the latest methodologies, instrumentation, and federal regulations on particulates.

TABLE IV. TIME SCHEDULE FOR COMPLETION OF PARTICULATES LABORATORY

Time Zero	Approval of particulates laboratory selection. Hire top scientist for laboratory. Choose architect for building.
2 Months	Architect completes drawing; request bids.
4 Months	Grant building-construction contract, order major equipment and clean-room module.
8 Months	Hire a scientist and senior technicians, order small laboratory equipment.

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TABLE IV (CONTINUED)

10 months	Building complete - install equipment. Run acceptance tests.
12 months	Begin sample investigations, hire remaining staff as required.
24 months	Laboratory capabilities fully developed.

After the completion of the architects' work, bids for construction may be requested. Four months after approval of the laboratory the construction contract for the building may be awarded. At this time the scientist in charge should order long-lead-time equipment such as electron optical instruments, x-ray diffractometers, and clean-room modules.

Interviewing of the skeletal staff for the laboratory (two senior technicians and one more scientist) should begin eight months after approval of the laboratory. Short-lead-time equipment such as optical microscopes and evaporators should be ordered at this point in the schedule.

With the completion of the building and clean rooms after 10 months, the equipment installation and testing period begins. Calibration and trial runs on the equipment using well-characterized particulate samples should be carried out during this period. After 12 months we anticipate that the laboratory should be able to handle some real-world specimens at a limited rate, with full capabilities taking an additional 12 months to develop. The number of staff to be added with time will be determined by the sample demand.

To expedite start-up, a contract should be arranged with a qualified laboratory to assist with placement of purchase orders, interface with an architect, co-ordinate recruiting, and train personnel.

6.2 Initial and Annual Costs

The costs listed below are valid as of February 29, 1980 but must be reassessed when this document is used. The initial cost of the laboratory and its equipment, excluding facility cost, and the annual operating cost for staff and supplies using GSA values (where available) are summarized below.

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Laboratory Building	24,000
Clean Fume Hoods (Option C) 14,000 Office furniture and equipment ~10,000	
Specimen Preparation Equipment	28,000
(Appendix C, Tables)	
Optical Microscopes	28,900
Stero microscopes (5) 4000 Polarizing microscopes (2) 15,200 Image shearing 9700	
X-Ray Diffractometer (Siemens)	119,300
ETEC or AMR - SEM with x-ray analyzer and digital beam control	190,000
Hitachi - 600 SCAN with x-ray analyzer and digital beam control	310,000
Darkroom Equipment (Appendix C, Table C-III)	31,300
Enlarger Logetronics Printer Plate processor Print processor and dryer	
Minicomputer	22,000
General Laboratory Supplies (Appendix C, Table C-II)	5,000
Electronic Maintenance Equipment (Appendix C, Table C-IV)	5,000
Total	$763,500

Annual Operating Costs of the Laboratory

A. Minimum Staff	
Personnel Costs (base salaries)	200,000
Personnel Benefits	48,000
Supplies & Equipment Maintenance	60,000
Total	308,000

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B. Optimum Staff

Personnel Costs (base salaries)	354,000
Personnel Benefits	80,000
Supplies & Equipment Maintenance	100,000
Total	534,000

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SECTION 7

SUMMARY AND RECOMMENDATIONS

7.1 Summary

A state-of-the art facility staffed and equipped to fully characterize the particulates found in the atmosphere is described in detail in the preceding sections of this report. The recommended conceptual design, if fully implemented, will achieve the objectives established by the EPA in commissioning this project. The broad purpose, of course, is to further the nation's long-range efforts to rectify the lack of reliable information about the exposure to asbestos and other particulates in nonoccupational settings and their health effects.

The current situation is not the result of a lack of effort on the part of EPA or other groups. To a large extent, it is a direct result of the ambiguity in the definition of asbestos and the intrinsic complexity and the inherent interdisciplinary nature of the analysis. Unfortunately, many of the projects sponsored to develop rigorous, robust methods for the analysis of asbestos and other particles have had limited success, and studies using recommended methods have been found wanting. Clearly a new approach is needed. Thus, establishment of a center of excellence within the EPA itself, competent to monitor and evaluate related activities in the various federal and private laboratories concerned with particulates analysis, is timely and well conceived.

7.2 Recommendations

Recommendations stemming from this evaluation and design project relating to the establishment of a particulates analysis facility, procedures for start-up, and initial development projects are listed below.

- Establishment of a federal laboratory specializing in the analysis of particulates should be of the highest priority.

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- The laboratory should be equipped with the best available instruments and staffed with highly qualified and experienced personnel.

- It should function as a center of excellence qualified to monitor and evaluate relevant efforts in any laboratory.

- An advisory panel should be established to aid in selecting programs, setting priorities, and evaluating progress but otherwise the laboratory should be autonomous.

- Peer review of the quality of the research program should be conducted under the auspices of the advisory panel.

- A substantial part of the program should be devoted to methods development and to providing a definitive analysis for critical samples; a minimum portion should be devoted to routine analysis.

- After start-up, funding should be provided to maintain state-of-the-art instrumentation as improvements become available.

- A contract should be arranged with a qualified laboratory to develop the necessary hardware and software to measure and interpret electron diffraction patterns from particulates.

- Initial research programs should be aimed at the development of suitable standards and reference samples, such as synthetic mixtures of asbestos and other materials, for general use.

- The laboratory should participate actively in round-robin studies sponsored by ASTM or other groups to establish the degree of reproducibility that can be achieved and should initiate such studies if necessary.

- The laboratory should screen and evaluate existing sample preparation techniques and develop new methods particularly suitable for AIA in both the SEM and STEM.

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- The laboratory should project the development of AIA methods, including more sophisticated computer software.

If the above recommendations are implemented, we believe the resulting laboratory will achieve a standard of excellence not previously available. The laboratory would then provide a major contribution to the development of knowledge about the occurrence and concentration of asbestos and other particulates in our environment so that one may assess their effects on human health, and ultimately the implementation of suitable regulations and control strategies.

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APPENDIX A

SPECIFICATIONS FOR THE SCANNING ELECTRON MICROSCOPE

SEM must meet or beat the following specifications:

Resolution	70Å
Magnification	12x to 200,000x @ 12 mm Working Distance Front panel LED readout of magnification and/or working distance with 1% reproducibility Automatic correction for changes in working distance and acceleration potential
Signals	Secondary electron, backscattered electron, and solid state STEM modes
Beam Current	10^{-12} - 10^{-6} amps
Accelerating Voltage	5-30 kV standard
Gun Type	Self biasing triode. Lanthanum Hexaboride cathode optional
Gun Alignment	External mechanical
Objective Aperture	3 externally selectable
Vibration Isolation	Pneumatic air suspension system
Display	2 CRT display with a 750 line resolution
Record	1 CRT with 2500 line resolution

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Scanning Rate	3 selectable viewing, 6 selectable record
Signal Processing	Multi-input selection, gamma control, invert, contrast and brightness meters
Vacuum System	Fully automatic, pushbutton operated, fully protected against power, water or vacuum failure
Pumps	Oil diffusion, liquid nitrogen trap
Valves	Fully interlocked gun isolation valve
Required Options	Automated stage Continuous KV Energy Dispersive X-ray system Digital scan generator Computer controlled zoom magnification unit

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APPENDIX B

SPECIFICATIONS FOR THE SCANNING TRANSMISSION ELECTRON MICROSCOPE

The recommended Hitachi-600 instrument (or any substitute) should have the following features.

A) Lanthanum Hexaboride electron source.

B) Electron Optics producing the following images and diffracting modes: STEM, TEM, SEM, BEI. All modes switch selectable without any other changes.

C) Two axis goniometer stage ±45°.

D) Si (Li) x-ray detector compatibility.

E) Magnification range 100-700,000X.

F) Contamination rate less than 0.1Å/min.

G) Automatic camera system for 30 sheets of film and automatic recording of operating conditions and film number.

H) LeMont Scientific Digital Beam Control.

I) Tracor Northern System 4000 for Energy Dispersive Analysis.

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APPENDIX C

Miscellaneous Laboratory Equipment

The special laboratory equipment required for specimen preparation, general laboratory supplies, darkroom operation, and maintenance of the electron microscopes and other facilities is listed in this section. The tables include the name and address of one of the major suppliers in the field and the most recent prices in current catalogs or as obtained by telephone inquiries.

Appendix Table C-I

Specimen-Preparation Equipment

Description and Use	Manufacturer	Total Price, Dollars
Mill - for grinding ores or rock samples grinds 18 kg/hr, 100 mesh, 115V 60 Hz #8-415	Fisher Scientific Co. 585 Alpha Drive Pittsburgh, Pa. 15238	480.00
Grinding Mill - hand powered for grinding wet or dry ores, minerals or clays. #8-450	"	58.50
Crusher - jar mill for crushing rocks and soil 2 tier, multi speed rollers, 115V 60 Hz Model 784AV #8-381-5	"	678.50

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Description and Use	Manufacturer	Total Price, Dollars
(2) Jars - for roller mill crusher, 1.1 liter capacity, #8-382B	"	165.60
(2) Agate mortar and pestle set for fine crushing, 95 mm OD #12-950B	"	226.00
U.S. Standard Sieves 8 Inch dia. (stainless)	Fisher Scientific Co. 585 Alpha Drive	
50 mesh (300 µm) #4-881-10T	Pittsburgh, PA 15238	42.75
100 mesh (150 µm) 4-881-10X		43.50
200 mesh (75 µm) 4-881-10BB		51.00
325 mesh (45 µm) 4-881-10EE		84.50
400 mesh (38 µm) 4-881-10FF		117.00
Sieve Cover stainless 4-887A	"	16.00
Stainless Deep Receiver 4-887B	"	30.25
Portable Sieve Shaker #3-910	"	255.50
Riffle Sampler $4-940	"	248.00
Riffle Drawers #4-940-5	"	26.00
Sonic Dismembrator, uses high frequency vibrations for uniform dispersion of particles Model 300 #15-338-40	"	1095.00
2.8 liter ultrasonic cleaner for homogeneous dispersion of particles in fluid before filtering. #15-337-15 and tank cover #15-337-21	"	162.75

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Description and Use	Manufacturer	Total Price, Dollars
Glass - 6 place, 47 mm, filter apparatus for vacuum filtering liquids containing particulates. #XX25 047 50	Millipore Corp. Order Service Dept. Bedford, Mass. 01730	1513.00
Miniature vacuum-pressure pump for 6 place filter apparatus. #XX61 000 00	"	231.00
Single all glass filter apparatus - 47 mm. For vacuum filtering liquids containing particulates. #XX15 047 00	"	103.00
Squeeze handle vacuum pump for single filtering apparatus. #1-070	Fisher Scientific Co. 585 Alpha Drive Pittsburgh, Pa. 15238	22.50
Millipore filters, 0.22 micron-pore size pkg. 100 discs #GSWP-04700	Millipore Corp. Order Service Dept. Bedford, Mass. 01730	28.70
Nuclepore filters, 0.2 micron clear, plain, regular, 47 mm, 100/Box. #4625-C25	Arthur H. Thomas P.O. Box 779 Philadelphia, Pa. 19105	27.50
SPI plasma prep low temperature asher for removing organic elements with partially ionized gases less than 50°C. SPI # 11001 and pair replacement pentode power tubes SPI #11021	Structure Probe, Inc. P.O. Box 342 West Chester, Pa. 19380	1843.00
SPI mechanical vacuum pump for low temperature asher. 55 liters per min, SPI #10303	"	615.00

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Description and Use	Manufacturer	Total Price, Dollars
Microtome, to cut thin sections of embedded filters or particles to any thickness from 1 to 50 microns. #12-601	Fisher Scientific Co. 585 Alpha Drive Pittsburgh, Pa. 15238	2160.00
Critical point dryer to preserve original shape and size of samples	Ladd Research Ind., Inc. P.O. Box 901 Burlington, Vermont 05401	1450.00
Sputter coater for coating SEM specimens with thin film of gold to prevent charging. Hummer V with vacuum pump, gold and carbon electrodes	Technics Inc. 5510 Vine St. Alexandria, VA. 22310	2995.00
DTM-Q quartz crystal digital thickness monitor for direct thickness readings in angstroms when coating specimens with Hummer V	Technics Inc. 5510 Vine St. Alexandria, VA 22310	2995.00
Hummer carbon evaporation accessory for carbon coating specimens for scanning and electron microscopy	"	850.00
Vacuum evaporator unit for coating filters to make carbon films with imbedded particles from the filters. Including accessories; #42000 carbon rod evaporation kit; $42100 carbon evap. shield; #40700 bell jar and #40750 cover; #40720 gasket; #61000 tilting rotary coater; #61200 variable voltage DC power supply	Ladd Research Ind., Inc. P.O. Box 901 Burlington, Vermont 05401	7735.00

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Description and Use	Manufacturer	Total Price, Dollars
Six unit counter for counting as many as six different types of particles. #2-670-13	Fisher Scientific Co. 585 Alpha Drive Pittsburgh, Pa. 15238	130.00
Cell counter for counting individual particles. #2-670-11	"	19.80
Refractive index liquids, 76 different liquids for identifying particles by phase contrast light microscopy	"	145.00
Electron diffraction measuring device #15-301	Baltzer Corporation 8 Sagamore Park Rd. Hudson, NH 03051	3600.00
Specimen preparation equipment and supplies		
	Total	$28,244.85

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APPENDIX TABLE C-II

General Laboratory Supplies

Description and Use	Manufacturer	Total Price, Dollars
Dumont Tweezers (12) #1101	Ernest F. Fullam	90.00
Silver Conducting Paint (5) #1481	P.O. Box 444 Schenectady, N.Y.	28.75
Dissecting Scissors (3) #5253	12301	14.55
Lettered Grids (500) #6220	"	85.00
Apiezon M Vac. Grease (2) #1267	"	35.50
Wiping Cloths #1149	"	20.25
POL Metal Polish (10) #1545	"	11.90
Effa Duster Refills (12) #1168	"	20.00
Effa Valve and Nozzle (2) #1167A	"	26.00
Desiccator Cabinet #2262	Ted Pella Inc.	160.00
Indicating Desiccant (26 lbs) #19660	P.O. Box 510	9.95
Carbon Dag (5) #16053	"	13.75
Carbon rods, 1-1/2 inches long CML Grade-YU-40, 100 rods/box #7529	Ultra Carbon Corp. P.O. Box 747 Bay City, Mich. 48706	56.00
7.6 liter ultrasonic cleaner for cleaning glassware #15-337-552 and tank cover #15-337-54	Fisher Scientific Co. 585 Alpha Drive Pittsburgh, Pa. 15238	384.00
	Total	$955.65

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Darkroom Equipment

Description and Use	Manufacturer	Total Price, Dollars
Electron microscope film and plate processor for developing and fixing negatives Model #EM 410-4A	Arkay Corp. 228 S. First St. Milwaukee, Wis. 53204	3125.00
Kodak royal print processor for developing and fixing prints. Model #417	Kadet Photo Supply Co. 1004 Fifth Ave. Pittsburgh, Pa. 15219	7990.00
Enlarger, for enlarging prints Omega, Pro-Lab D5V	"	805.00
Omega, EL-Omegar, 75 mm lens	"	32.50
Omega, O Micron-EL, 105 mm lens	"	168.95
Omega, O Micron-EL, 135 mm lens	"	234.95
Arkay speed dodge contact printer, Model AP-18	"	520.00
Thomas duplex safelight	"	175.00
Mark IV contact printer, with enlarger attachment, for gang printing negatives of varying contrasts	Logetronics, Inc. 7001 Loisdale Rd. Springfield, VA. 22150	18,000.00
Stainless steel developing tanks for processing SEM Polaroid negatives (3) #2606	Ted Pella, Inc. P.O. Box 510 Justin, Calif. 92680	93.00
Stainless steel covers for developing tanks (3) #2612	"	36.00

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APPENDIX TABLE C-III (CONTINUED)

Description and Use	Manufacturer	Total Price, Dollars
Stainless steel film washer #261-58	"	77.50
Film clip hangers (50) #268-7	"	15.00
	Darkroom Supplies	
	Total	$31,272.90

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APPENDIX TABLE C-IV

Electronic Maintenance Equipment

Description and Use	Manufacturer	Total Price, Dollars
Tektronix oscilloscope T912 with storage	Tektronix 3834 Northern Pike Monroeville, Pa. 15146	1545.00
Option 01 differential input for checking wave forms of electronic circuits	"	50.00
H.P. electronic counter, 5300B measurement system main frame	Hewlett Packard 111 Zeta Drive Pittsburgh, Pa. 15238	460.00
Universal counter-timer module 5308A used to check pulse counts from x-ray counters	"	450.00
H.P. digital voltmeter:	"	
34750A display		750.00
34702A multimeter		325.00
34111A D.C. Hi voltage probe for measuring voltages		75.00
Logic probe LP-1 to check integrated circuit pulses at specific points	Continental Specialties Corp. Div. of Marlow Assoc., Inc. 144 Cypress Drive Pittsburgh, Pa. 15241	45.00

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APPENDIX TABLE C-IV (CONTINUED)

Description and Use	Manufacturer	Total Price, Dollars
Logic Monitor LM-2 to check integrated circuit pulses from a complete chip or unit	"	125.00
Jensen master industrial electronic technician tool kit JTK-5 complete set of hand tools	Jensen Tool and Alloys 1230 S. Priest Drive Tempe, Arizona 85281	234.00
Weller controlled-output soldering station WTCPN for critical soldering	CAM RPC Industrial Electronics 620 Alpha Drive Pittsburgh, Pa. 15238	50.00
	Electronic maintenance equipment	
	Total	$4109.00

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APPENDIX D

PERSONNEL TRAINING AND DEVELOPMENT

Recommended courses on Light and Electron Optical Methods of Particulate Analysis.

Optical Microscopy

McCrone Research Inst. Inc., Chicago, Ill.
A 1-week course of phase contrast and polarized light microscopy.

Electron Microscopy

The instrument manufacturers mentioned in this report provide training courses of about 1 week duration, approximately four times per year, in the operations and maintenance of electron microscopes.

The Electron Microscopy Society of America and the Materials Analysis Society present 1- and 2-day workshops on various methods of electron microscopy in connection with their annual meetings.

Short courses of 1 or 2 weeks duration are presented regularly by:

Lehigh Univ.	Dept. of Metallurgy and Materials Engineering, Bethlehem, Pa.
Duke Univ.	Electron Microscope Institute, Research Triangle Park, N.C.
George Washington Univ.	Dept. of Anatomy, Washington, D.C.
Orange County Community College	Middletown, N.Y.

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APPENDIX D (CONTINUED)

RECOMMENDED DEVELOPMENT COURSE FOR PERSONNEL

McCrone Research Institute

MICROSCOPICAL IDENTIFICATION OF ASBESTOS AND OTHER SMALL PARTICLES

Course Curriculum

Monday

Lecture:	Physical optics, the polarizing microscope and its parts, Köhler illumination.
Laboratory:	Setting up the microscope and illumination system, micrometry.
Lecture:	Crystal morphology, Refractive index, crystal optics, (one polar only).
Laboratory:	Study of crystal morphology, measurement of refractive indices for isotropic and anisotropic substances.

Tuesday

Lecture:	Crystal optics (crossed polars).
Laboratory:	Use of the Michel-Levy chart, birefringence compensation.
Lecture:	Asbestos: sources, uses, chemistry, crystallography, dispersion staining.
Laboratory:	Study of known chrysotile and amphiboles.

Wednesday

Lecture:	Characterization of asbestos types and interfering substances by dispersion staining.
Laboratory:	Study of known and unknown asbestos samples.

Thursday

Lecture:	Phase microscopy.
Laboratory:	Phase microscopy. Preparation and counting of membrane filters by OSHA method.
Lecture:	Other methods of characterizing asbestos, i.e., SEM, TEM, XRD, DTA.
Laboratory:	Study of unknown samples containing asbestos, e.g., mortars, air samples and talcs.

Friday

Lecture:	Sampling methods and identification of small particles on membrane and high volume filters.
Laboratory:	Characterization of filter samples.

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